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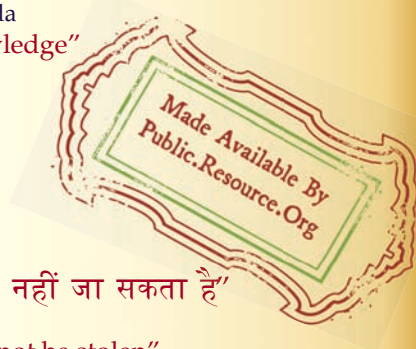
IS 5278 (1969): Dicofol, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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Indian Standard
SPECIFICATION FOR
DICOFOL, TECHNICAL

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Indian Standard

SPECIFICATION FOR DICOFOL, TECHNICAL

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Indian Standard

SPECIFICATION FOR DICOFOL, TECHNICAL

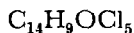
0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 September 1969, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

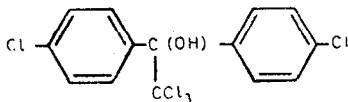
0.2 Dicofol, technical, is used in preparing acaricidal formulations for the control of acarine pests of fruit vegetable crops.

0.2.1 Dicofol is the common name accepted by the International Organization for Standardization for the pesticidal chemical containing 2,2,2-trichloro-1,1-di-(4-chlorophenyl) ethanol as its active ingredient. The structural and empirical formulæ and the molecular weight of this compound are indicated below:

Empirical Formula



Structural Formula



Molecular Weight

370.5

0.3 Taking into consideration the views of producers, consumers, testing authorities and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the manufacturing and trade practices followed in the country in this field.

0.4 This standard is one of a series of Indian Standards on pesticides and their formulations.

0.5 This standard contains clauses **C-2.3** and **C-3.4** which call for an agreement between the purchaser and the vendor.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for dicofol, technical, employed in the preparation of acaricidal formulations.

2. REQUIREMENTS

2.1 Description—The material shall be in the form of a dark viscous liquid. It shall be free from extraneous impurities and modifying agents.

2.2 The material shall comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR DICOFOL, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix (4)	Indian Standard (5)
(1)	(2)	(3)	(4)	(5)
i)	Active ingredient content (see 0.2.1), percent by weight, <i>Min</i>	82.0	A	—
ii)	Moisture content, percent by weight, <i>Max</i>	0.05	—	IS: 2362-1963*
iii)	Acidity (as H_2SO_4), percent by weight, <i>Max</i>	0.3	B	—

*Method for determination of water by the Karl Fischer method.

3. PACKING AND MARKING

3.1 Packing—The material shall be packed in containers made of steel lined with baked phenolic or baked epoxy phenolic terne-plated steel or anodized aluminium drums.

3.2 Marking—The containers shall be securely closed and shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer;
- Date of manufacture;
- Batch number;
- Net weight of contents;
- Active ingredient content; and
- The minimum Cautionary Notice worded as under:

‘KEEP AWAY FROM FOODSTUFFS AND ANIMAL FEEDS. DESTROY THE EMPTY CONTAINERS.’

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing the representative samples of the material and criteria for conformity shall be as prescribed in Appendix C.

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices given in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[Table 1, Item (i)]

DETERMINATION OF ACTIVE INGREDIENT CONTENT

A-1. REAGENTS

A-1.1 **Ethyl Alcohol** — 95 percent.

A-1.2 **Ethanolic, Potassium Hydroxide** — 0.5 N.

A-1.3 **Concentrated Nitric Acid** — analytical reagent grade conforming to IS: 264-1968†.

A-1.4 **Ferric Alum Indicator** — Dissolve 350 g of ferric ammonium sulphate crystals in 700 ml distilled water, heating gently if necessary. Add 50 ml of concentrated nitric acid, dilute to 1 litre with distilled water and mix thoroughly.

*Specification for water, distilled quality (*revised*).

†Specification for nitric acid (*revised*).

A-1.5 Nitrobenzene, Technical Grade**A-1.6 Standard Silver Nitrate, Solution**—0.1 N.**A-1.7 Standard Ammonium Thiocyanate, Solution**—0.1 N.

A-1.8 Phenolphthalein Indicator Solution—Dissolve 0.5 g of phenolphthalein powder in 100 ml of 95 percent ethyl alcohol.

A-2. PROCEDURE

A-2.1 Weigh accurately about 0.5 g of the sample, weighed to 0.0001 g, and transfer to a 300 ml Erlenmeyer flask. Add 50 ml of the standard ethanolic potassium hydroxide solution to the sample. Place the flask on a hot-plate and connect the neck of the flask to water-cooled condenser, using 3 to 4 drops of 95 percent ethyl alcohol as a seal. Boil the solution under gentle reflux for 90 minutes. At the end of this time, stop heating and allow the flask to cool slightly, rinse the condenser with 25 ml of 95 percent ethyl alcohol. Disconnect the flask and rinse the tip of the condenser with 95 percent ethyl alcohol, adding the rinsings to the flask.

A-2.2 Drain the liquid contents of the flask into a 400-ml beaker, add 50 ml of 95 percent ethyl alcohol to the flask. Swirl the flask, and again drain the liquid contents into the 400-ml beaker. Add approximately 100 ml of distilled water to the flask, swirl and add the contents of the flask to the 400-ml beaker by stirring. Rinse the flask with small portions of distilled water and add the rinsings to the 400-ml beaker by stirring. Continue rinsing the flask until there is a total volume of approximately 300 ml in the beaker.

A-2.3 Place the beaker on a steam-bath until all of the alcohol has been evaporated. Cool the beaker in a water-bath and add 2 to 3 drops of standard phenolphthalein indicator solution, and sufficient 1:1 nitric acid to turn the indicator colourless. Add an additional 10 ml of 1:1 nitric acid and 50.0 ml of the standard silver nitrate solution. Thoroughly mix the contents of the beaker. Add 5 ml of ferric alum indicator solution, 5 ml of nitrobenzene to the beaker, stir vigorously and titrate the excess silver nitrate with the standard ammonium thiocyanate solution with vigorous stirring, until the appearance is of a faint but permanent pink end-point.

A-3. CALCULATION

A-3.1 Active ingredient, percent content =

$$\frac{(\text{ml AgNO}_3 \text{ for sample} - \text{ml AgNO}_3 \text{ for blank})}{\text{weight of sample (grams)}} \times N \times 12.351$$

NOTE—Run a blank determination through all steps of the procedure using all reagents except the material to be analyzed.

APPENDIX B

[Table 1, Item (iii)]

DETERMINATION OF ACIDITY

B-1. REAGENTS

B-1.1 Acetone

B-1.2 Methyl Red Indicator Solution — aqueous, one percent (w/v).

B-1.3 Standard Sodium Hydroxide Solution — 0.02 N.

B-1.4 Standard Hydrochloric Acid — 0.02 N.

B-2. PROCEDURE

B-2.1 Weigh accurately about 10 g of the material and dissolve it in 25 ml of acetone. Solution may be effected by gently warming, if necessary. Add 75 ml of water and titrate immediately with the standard sodium hydroxide solution using methyl red as the indicator.

B-2.2 Carry out a blank determination using 25 ml of acetone diluted with 75 ml of water.

B-3. CALCULATION

B-3.1 Acidity (as H_2SO_4), percent by weight = $\frac{4.9 (V - v) N}{W}$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N = normality of the standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

B-3.1.1 In case the blank determination shows an alkaline reaction, neutralize with the standard hydrochloric acid and calculate the acidity as follows:

Acidity (as H_2SO_4), percent by weight = $\frac{4.9 (VN_1 - vN_2)}{W}$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

N_1 = normality of the standard sodium hydroxide solution,

- v = volume in ml of the standard hydrochloric acid required for the blank determination,
 N_2 = normality of the standard hydrochloric acid, and
 W = weight in g of the material taken for the test.

APPENDIX C

(Clause 4.1)

SAMPLING OF DICOFOL, TECHNICAL

C-1. GENERAL PRECAUTIONS

C-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be taken in an exposed place.

C-1.2 The sampling instrument shall be clean and dry when used.

C-1.3 Proper precautions shall be taken while drawing samples since the material is toxic.

C-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the receptacles for samples from adventitious contamination.

C-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or by any other suitable means so as to bring all portions into uniform distribution.

C-1.6 The sample shall be placed in suitable, clean, dry and air-tight, sample receptacles. The cap or lid of the sample receptacle shall be lined with tin-foil.

C-1.7 The sample receptacles shall be of such a size that they are almost, but not completely, filled by the sample.

C-1.8 Each sample receptacle shall be sealed air-tight after filling and marked with full details of sampling, the date of manufacture, name of the manufacturer and other particulars of the consignment.

C-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature. The sample shall be heated at 70°C (80°C *Max*) before removing for analysis.

C-2. SCALE OF SAMPLING

C-2.1 Lot—All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or is known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

C-2.1.1 Samples shall be tested separately for each lot for ascertaining the conformity of the material to the requirements of the specification.

C-2.2 The number (n) of containers to be selected from the lot shall depend on the size (N) of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(Clauses C-2.2 and C-2.3)

NO. OF CONTAINERS IN THE LOT	NO. OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
3 to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
Over 110	10

C-2.3 These containers shall be selected at random from the lot. To ensure the randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be used:

Starting from any container, count all the containers as 1,2,3,....., up to r and so on, in one order, where r is equal to the integral part of N/n ; N being the number of containers in the lot, and n the number of containers to be selected (Table 2). Every r th container thus counted shall be withdrawn until the requisite number of containers is obtained from the lot to give samples for test.

C-3. TEST SAMPLES AND REFEREE SAMPLES

C-3.1 Before drawing the test sample, thoroughly mix the contents of each container selected by shaking or by any other suitable means. Draw small portions of the material from different parts of each container selected (Table 2). The total quantity of the material drawn from each container shall not be less than 100 g.

C-3.2 Mix thoroughly all portions of the material drawn from the same container. Out of these portions, small but equal quantity shall be taken for each selected container and shall be well mixed together so as to form a composite sample of not less than 150 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

C-3.3 The remaining portions of the material from each container (after a small quantity needed for the formulation of the composite sample has been taken out) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried sample receptacles which are then sealed air-tight, and labelled with all the particulars of sampling given under **C-1.8**. The material in each such sealed sample receptacle shall constitute a test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each container selected (*see* Table 2). One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

C-3.4 Referee Samples — Referee samples shall consist of the composite sample (**C-3.2**) and a set of individual test samples (*see* **C-3.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the two.

C-4. NUMBER OF TESTS

C-4.1 Tests for the description (*see* **2.1**) and determination of the active ingredient content shall be conducted individually on each of the samples constituting a set of individual test samples.

C-4.2 Tests for the determination of remaining characteristics, namely, moisture and acidity shall be conducted on the composite sample (*see* **C-3.2**)

C-5. CRITERIA FOR CONFORMITY

C-5.1 A lot shall be declared as conforming to this specification when:

- a) each of the samples tested for description satisfies the requirements given in **2.1**;
- b) each of the test results for active ingredient content satisfies the requirement as specified in Table 1; and
- c) the test results on the composite sample satisfy the requirements for the remaining characteristics specified in Table 1.

C-5.1.1 If one or more test results do not satisfy the requirements for active ingredient content, the following procedure shall be adopted

for determining the conformity of the material for this characteristic:

Calculate the mean and range as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of test samples}}$$

$$\text{Range } (R) = \text{Difference between the maximum and the minimum value of the test results.}$$

The requirement for active ingredient content shall be considered conforming to this specification if $\bar{X} - 0.6R > 82.0$.

(Continued from page 2)

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